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PROFESSIONAL PAPER.

THE PRODUCTION AND FERTILIZER VALUE OF CITRIC-SOLUBLE PHOSPHORIC ACID AND POTASH.

By WM. H. WAGGAMAN,
Scientist in Investigation of Fertilizer Resources.

INTRODUCTION.

The extraction of potash from silicate rocks or the rendering of this alkali soluble in water has been and probably will continue to be for a long time the object of numerous investigations.

Ross[1] has investigated many of these processes and discussed several in some detail. For convenience he divides them into three classes, as follows: (1) Processes which yield potash as the only product of value; (2) processes which yield potash and some other salable material as a by-product; (3) processes in which two or more operations are combined in one, yielding a fertilizer containing two or more of the constituents, potash, phosphoric acid, and nitrogen. He describes two methods for obtaining potash from feldspar by treating mixtures of that mineral and lime, collecting the potash thus liberated, and using the residue for the manufacture of cement. The potash obtained by these processes, however, is in the form of oxide or hydroxide, and is therefore more valuable for other purposes than for the manufacture of fertilizers. Ross also tried heating together feldspar and lime with the addition of phosphate rock, but found that the latter substance did not enter into the reaction, there being no increase in the quantity of potash thus obtained over that produced by the ignition of feldspar and lime alone.

The production in a single operation of available phosphoric acid and potash from insoluble minerals, however, presents possibilities which are particularly attractive, and several processes have been devised to accomplish this end. It is the purpose of this paper first to discuss these existing methods and then to describe a process recently devised in this laboratory for rendering the phosphoric acid and potash in a mixture of phosphatic limestone and feldspar "citric soluble."

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[1] Jour. of Ind. and Eng. Chem., 5, No. 9, pp. 725–729 (1913).

EARLIER METHODS.

There are four[1] recorded processes for making a phosphoric acid-potash fertilizer from phosphate rock and feldspar. In chronological order they are as follows:

A method devised by Charles Bickell[2] in 1856, which consists in heating in a reverberatory furnace to a light redness for two hours an intimate mixture of 1 part feldspar, 0.5 part phosphate of lime, and 3 or 4 parts of air-slaked lime. Bickell claims that both phosphoric acid and potash in available forms are obtained by this treatment.

This experiment was repeated in this laboratory, using feldspar, Tennessee phosphatic limestone, and calcium carbonate in the following proportions:

	Per cent.
Feldspar (13.7 per cent K_2O)	22.2
Phosphatic limestone (23 per cent P_2O_5)	11.1
Calcium carbonate	66.7

Assuming that the carbon dioxide present in the calcium carbonate and phosphatic limestone was the only substance volatilized during the process, the product should have contained after ignition 4.38 per cent potash (K_2O) and 3.4 per cent phosphoric acid (P_2O_5). Analysis of the residue, however, gave the following results:

	Per cent.
K_2O:	
Total	1.94
Water soluble	.18
P_2O_5:	
Total	3.62
Citric soluble	1.40

These results show that over 44 per cent of the potash present in the mixture was volatilized upon ignition, and of that which remained in the residue only 9 per cent was water soluble. While none of the phosphoric acid was volatilized, less than 39 per cent of the total amount present was soluble in a 2 per cent solution of citric acid (the method usually employed for determining the availability of phosphoric acid in basic slag).

The second process for the manufacture of a phosphate-potash fertilizer from feldspar and phosphate rock was devised by Frederick Klett[3] in 1865. It consists of heating to redness for five hours an intimate mixture of one part feldspar, two parts carbonate of lime, one part phosphate rock, and adding for each part of K_2O in the feldspar two parts of calcium fluoride. It is claimed that a soluble silicate of lime and potassium phosphate are thus obtained. In view of the fact that the percentage of potash in the mixture is relatively small and that the time of heating is very long, it is hardly likely that the value of the product would cover the cost of manufacture. Moreover, the claim that phosphate of potash is formed in the operation is apparently not justifiable. It was thought advisable, however, to test this process also. A mixture of the following composition was made up and ignited in a muffle furnace for five hours at red heat:

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[1] Since transmitting this manuscript several other processes have been devised. The author regrets that it is impracticable to consider these methods in the present paper.

[2] U. S. Patent No. 16111 (1856).

[3] U. S. Patent No. 49891 (1865).

	Per cent.
Feldspar (13 per cent K_2O)	24.21
Phosphate rock (32.8 per cent P_2O_5)	24.21
Calcium carbonate	48.42
Calcium fluoride	3.15

The slightly sintered product of this mixture was finely ground and analyzed both for potash and phosphoric acid. If carbon dioxide were the only volatile substance formed by heating the above mixture the final product should have contained 4 per cent of K_2O and 10.09 per cent of P_2O_5. Actual analysis of the material, however, gave the following results:

	Per cent.
K_2O:	
Total	0.60
P_2O_5:	
Total	10.51
Citric soluble	4.15

Here again, as in Bickell's process, the potash nearly all volatilized, while less than one-half of the phosphoric acid present in the residue is citric soluble.

The third process for rendering the phosphoric acid and potash of rocks available for fertilizer purposes is that of Coates,[1] which consists in adding to the sterlized rock mixture certain microorganisms that effect the breaking down of the rock minerals. It is understood that the material thus prepared is being tried out experimentally by actual field tests, the results not yet having been reported.

In 1912 Haff[2] devised a process for making potassium phosphate from a mixture of feldspar and phosphate rock. The method is based on the fact that at high temperatures and in the presence of silica and a nonvolatile base, both potash and phosphoric acid are volatilized. Haff claims that 95 per cent of the potash and phosphoric acid of natural rocks can be driven off at a temperature of 2,000° C. and collected by passing the fumes through scrubbing towers. While this method has not been tried out in this laboratory, the cost of maintaining the high temperature necessary for the decomposition of the minerals and the expense of collecting and recovering the potash and phosphoric acid thus violatilized make it very doubtful if this process is commercially practicable.

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[1] U. S. Patent No. 947795 (1910).

[2] U. S. Patent No. 1018186 (1912).

THE FUSION OF FELDSPAR AND PHOSPHATE ROCK.

The solubility of phosphates in certain organic solutions has for a long time been regarded as a test of their agricultural availability. The nature and strength of the organic solvent used differs in various countries, and since each process is based on an arbitrary standard, it can only give corresponding results when conditions are the same. But in spite of the fact that none of these methods is founded upon a strictly scientific basis, it is generally thought that phosphates soluble in such solutions are under soil conditions more active than those which do not dissolve in the same mediums.

The beneficial effect of the phosphoric acid in finely ground steamed bone is unquestioned, and although little of it is water soluble, a neutral solution of ammonium citrate will dissolve from 12 to 31 per cent of the acid, depending on the temperature of the solution and the time of contact.[1] It is also an indisputable fact that excellent results have been obtained by the use of basic slag as a fertilizer, and it is claimed that these results are commensurable with the amount of citric soluble phosphoric acid present in the material.

It has therefore become customary to regard citrate or citric soluble phosphoric acid as having a commercial value nearly equal to that of water soluble phosphate.[2] While this is true of phosphates, the same view is not taken of potash-bearing substances, since practically all of the potash carriers used in agriculture are water soluble. The potash in the ordinary soil minerals is almost entirely insoluble in water, and but slightly soluble in the mineral acids, but if the potash present could be converted into a citric soluble form there seems to be no reason why it should not be considered as available to crops as citric soluble phosphoric acid.

During some investigations carried on in this laboratory on the possibilities of rendering the slags from the iron and steel industries available for fertilizer purposes,[3] attempts were made to fuse together mixtures of feldspar and phosphatic limestone with a view to obtaining both the potash and phosphoric acid present in an "available" form. Mixtures containing various proportions of these two substances were ignited at temperatures ranging from 1,200° to 1,400° C., but only viscous fusions were obtained and the potash and phosphoric acid in none of these were soluble in 2 per cent citric acid, the solution conventionally employed to determine the availability of phosphoric acid in basic slag.

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[1] Huston, H. A., 32d Annual Report Indiana State Board of Agriculture, p. 230 (1883); Wiley, H. W., Principles and Practice of Agricultural Analysis, vol. 2, p. 47; Wheeler, H. J., Manures and Fertilizers, p. 172 (1913).

[2] A report of a conference of the experiment stations of New York, New Jersey, and New England, Mar. 1, 1911, indicates that citrate soluble phosphoric acid has about nine-tenths of the fertilizer value of water soluble phosphoric acid.

[3] Bul. 95, Bureau of Soils, U. S. Dept. of Agr., 1912.

The failure to obtain liquid fusions was at the time attributed to the absence of iron or manganese, or both, in the mixtures, since it is well known that these elements impart fluidity to slags in the manufacture of iron and steel. Subsequent experiments have proved that such was the case, for on the addition of small quantities of these two elements to certain mixtures of feldspar and phosphatic limestone liquid fusions were obtained, and these were found to contain both phosphoric acid and potash in a citric soluble form.

In Table I is given the composition of the materials used in the experiments.

TABLE I.—*Composition of feldspar and phosphatic limestone used.*

Material.	SiO_2.	Al_2O_3.	CaO.	K_2O.	Na_2O.	P_2O_5.	CO_2.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Feldspar	65.7	18.4	....	13.7	2.2	....	....
Phosphatic limestone	32.4	....	37.1	....	....	20.0	10.5

The above materials were mixed in various proportions and small amounts of hematite and manganese dioxide were added. Each mixture was then placed in a graphite crucible and heated in a muffle furnace until fusion took place. The melts were then cooled, finely ground, and analyzed for phosphoric acid and potash (both total and citric soluble).

It was found that while citric soluble potash can be readily obtained by heating various mixtures of feldspar and phosphatic limestone over a wide range of temperatures, the limits within which the maximum yields of citric soluble phosphoric acid are obtained are quite narrow, both in respect to the proportion of the ingredients in the mixture and the length of time of heating.

The percentages of the ingredients used in the various mixtures before ignition and the composition of the melts are given in Table II.

TABLE II.—*Proportions in which ingredients were mixed and analyses of melts obtained.*

Sample No.	Amount of material used.				Properties of fusion.	Analysis of melts.			
	Feldspar.	Phosphatic limestone.	Fe$_2$O$_3$.	MuO$_2$.		P$_2$O$_5$ soluble in citric acid.	Total P$_2$O$_5$.	K$_2$O soluble in citric acid.	Total K$_2$O.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*		*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
25 S	42.8	42.8	8.6	5.7	Slightly viscous	2.12	9.76	6.56	6.82
28 S	37.5	50.0	7.5	5.0	do	1.50	11.40	5.32	5.68
33 S	46.8	37.5	9.4	6.3	Liquid	7.22	7.21	6.50	6.48
34 S	37.5	42.5	12.5	7.5	do	5.80	[1] 9.13	4.50	5.26

[1] Not determined by analysis, but calculated from the quantity of feldspar added.

Table II shows that nearly all of the potash present after fusing the various mixtures was soluble in 2 per cent citric acid, but in only one case (33S) was the total phosphoric acid present after ignition soluble in this same medium.

Further investigation showed that both the quantity and solubility of the phosphoric acid remaining in this melt was greatly influenced by the temperature and length of time of heating. In order to test the effect of these two factors on the composition and nature of the melt, this mixture (33S) was heated for various periods of time and at several different temperatures. The melts thus obtained were cooled, ground, and analyzed. The results of these analyses are given in Table III.

TABLE III.—*Properties and analyses of melts obtained by heating together phosphatic limestone and feldspar with small amounts of hematite and manganese dioxide.*

Sample No.	Temperature of melt.	Time of heating.	Properties of fusion.	P$_2$O$_5$.		K$_2$O.	
				Citric soluble.	Total.	Citric soluble.	Total.
	° C.	*H. m.*		*Per cent.*	*P. ct.*	*Per cent.*	*Per cent.*
33 SA	1,200	0 20	Viscous	1.82	8.50	5.98	6.76
33 SB	1,400	0 40	Liquid	7.22	7.21	6.50	6.48
33 SC	1,400	1 40	Less liquid	2.50	3.76	Lost.	5.48
33 SD	1,400	4 40	Viscous	1.20	2.88	Not determined.	Not determined.

It is shown in Table III that when the mixture was heated to 1,200° C. for about 20 minutes the fusion was not complete, and only a little more than 21 per cent of the total phosphoric acid present was citric soluble; over 88 per cent of the potash, however, was soluble in the same medium. Upon raising the temperature to 1,400° C. and maintaining it there for 20 minutes the fusion became quite fluid, and, although small amounts of potash and phosphoric acid were lost through volatilization, the remainder of these ingredients was entirely soluble in 2 per cent citric acid. After heating the mixture for one hour longer at the same temperature almost 50 per cent of the phosphoric acid and more than 15 per cent of the potash were volatilized, and upon heating for three hours more the phosphoric acid was still further reduced.

The sample richest in citric soluble phosphoric acid and potash (33S B) was submitted to a microscopic examination by Mr. W. H. Fry of this bureau. It was found to be isotropic and possessed all the external characteristics of a glass.

This was to be expected, however, since the melt was cooled too rapidly to allow of its crystallization.

SOLUBILITY OF THE POTASH OF THE SLAG IN WATER SATURATED WITH CARBON DIOXIDE.

The fact that the phosphoric acid of basic slag is fairly soluble in water saturated with carbon dioxide is taken as an added proof of its availability under soil conditions. It was thought advisable, therefore, to test the solubility of the potash in the slag product (33SB) in this same medium, comparing this solubility with that of the potash in feldspar.

Considerable work has been done on the so-called solubility of orthoclase in water and in various other solvents.[1] It is recognized, however, that this mineral has no definite solubility in water, but the dissolved material undergoes practically complete hydrolysis or decomposition, the amount of this decomposition being considerably affected by the fineness of the mineral, the method of grinding it (whether wet or dry), the quantity and temperature of the water used, and the length of time the water is allowed to act.

In Table IV the apparent solubility of feldspar in pure water and in water saturated with carbon dioxide as determined by several investigators is given. Few of these results are comparable, owing to the different conditions under which the experiments were conducted, but they are of interest in showing what widely divergent results are obtained by varying these conditions.

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[1] Roger Brothers, Am. Jour. Sci. and Arts. 2, 401 (1848) ; Daubrer, A., Études Synthétiques de Géologie Expérimentales, pp. 268–275 (1879) ; Clarke, F. W., Jour. Am. Chem. Soc., 20, 739 (1898) ; Lemberg Inaugural Dissertation Dorkat (1877) ; Cameron and Bell, Bul. 30, Bureau of Soils, U. S. Dept. of Agriculture (1905) ; Cushman and Hubbard, Bul. 28, Office of Public Roads, U. S. Dept. of Agriculture (1907). See also Cameron, Proceedings Eighth International Congress of Applied Chemistry, New York, 1912, Vol. XV, p. 43 et seq.

TABLE IV.—*Solubility of feldspar in water and in water saturated with CO_2, as determined by various investigators.*

Investigator.	Amount of feldspar used.	Amount of water used.	Time of contact.	Method of treatment.	Amount of K_2O dissolved from feldspar.	Amount of K_2O in solutions.
	Grams.	*Grams.*			*Per cent.*	*P. p. m.*
Daubrer	3,000	5,000	192 hours	Triturated in revolving cylinder.	0.420	2,520.0
Do	2,000	(CO_2) 3,000	240 hours	do	.013	90.0
Cameron and Bell	2	1,000	14 months.	Allowed to stand in contact with water.	.085	1.7
Do	2	(CO_2) 1,000	do	do	.125	2.5
Cushman and Hubbard.	25	100	Not given.	Mineral ground while dry.	[1] .100	[1] 250.0
Do	25	100	do	Mineral ground wet.	[1] 1.280	[1] 3,200.0

[1] Total residue.

In the experiments for comparing the solubility of the potash in 33SB with that of feldspar, a large quantity of the slag product was first prepared and ground to pass a 200-mesh screen. On analysis this material showed the following composition:

	Per cent.
K_2O citric soluble	5.14
K_2O total	5.43
P_2O_5 citric soluble	6.32
P_2O_5 total	6.31

Two samples of this slag of 25 grams and 0.25 gram, respectively, and the same quantities in weight of feldspar (ground equally fine) were placed in platinum-lined brass cylinders, 100 cubic centimeters of water were added to each and carbon dioxide under an average pressure of 1⅛ atmospheres was passed through the solutions for one week. At the end of that time the amount of potash dissolved from each sample was determined by analysis of the solutions. The results of these analyses are given in Table V.

TABLE V.—*Solubility of the potash in the slag product (33SB) in water saturated with carbon dioxide, compared with that of the potash in feldspar.*

Material used.	Amount of material used.	Amount of water employed.	Potash dissolved.	
			Potash in solution.	Percentage of total potash in sample.
	Grams.	*C. c.*	*P. p. m.*	
Feldspar	25.00	100	127.5	0.36
Do	.25	100	4.7	1.37
Slag (33SB)	25.00	100	150.0	1.11
Do	.25	100	11.9	8.76

In the case where large samples (25 grams) of slag and feldspar were used the resulting solutions differed but little in respect to their potash content, but considering the fact that the sample of slag contained less than one-half as much potash as the feldspar, the percentage of potash dissolved from the former was nearly four times as great. Moreover, while no quantitative determinations were made of the total solids in solution, the amount dissolved from the slag was apparently many times greater than that dissolved from the feldspar.

In the case where only 0.25 gram samples of the two substances were used the slag yielded a solution nearly three times as strong (in respect to potash) as the feldspar, and when the quantity of potash in the two substances is considered the percentage dissolved from the slag was nearly seven times greater.

RESULTS OF POT TESTS WITH SLAG FERTILIZER.

Although the solubility of the potash and phosphoric acid in the slag product was indicative of its agricultural value it was thought advisable to test its merits by actually growing plants in soils treated with this material, and comparing their growth with that of plants grown under similar conditions in the same soils untreated and treated with well-known potassic and phosphatic fertilizers.

The experiments were conducted by Mr. J. J. Skinner of this bureau. The wire-basket method described in Circular 18, Bureau of Soils, was employed, using wheat seedlings.

Three types of soil were used, namely, the Carrington silt loam from Wisconsin, the Hagerstown loam from Pennsylvania, and the Volusia silt loam from New York. These soils are described in Bulletin 96 of this bureau as follows:

> The Carrington silt loam consists of a dark-brown to black silt loam, having an average depth of about 12 inches. The subsoil is a yellowish-brown to pale-yellow silty clay loam or silty clay. The topography is mainly level to undulating. The soil represents a residual stratum derived from glacial till. The type is admirably adapted to the general farm crops, including wheat, corn, oats, barley, rye, flax, and grass.
>
> The Hagerstown loam is a brown or yellow loam averaging about 12 inches in depth. The subsoil is a yellow or reddish clay loam to a depth of 24 inches, but frequently grades into a stiff, yellowish-red clay. The type occupies rolling valley land, and is derived from the weathering of pure limestone. This is typical corn soil. It is one of the best general farming types in the eastern States, and is used for corn, tobacco, wheat, grass, and apples.
>
> The soil of the Volusia silt loam, to an average depth of 8 inches, is a gray to brown silt loam. The subsoil to a depth of 2 feet is a light-yellow silt loam, at which point mottlings of gray or drab are encountered. Both soil and subsoil

contain a high percentage of flat fragments of shale and sandstone, ranging from 1 or 2 inches to a foot or more wide. In addition, a considerable quantity of finely divided shale fragments is found in both soil and subsoil. The subsoil usually rests at varying depths below 18 inches on beds of shale or sandstone rock. The type is derived from the weathered products of the shale and sandstone, reworked by glaciation and slightly modified by extraneous glacial material. It occupies rolling and hilly land and is frequently interrupted or bordered by steep slopes not suited to agricultural purposes. The Volusia silt loam where properly cultivated is a good soil for timothy and small grains. In the eastern part of the region where it occurs it lies at too high an elevation to be well adapted to corn. In this region buckwheat and potatoes are grown to advantage.

Each of the above soils was treated with applications of the slag fertilizer, and wheat seedlings were planted. The plants were grown for a period of three weeks and then weighed and compared with those grown under similar conditions in untreated soil and in soil treated with other forms of potash and phosphatic fertilizers. Since the Volusia silt loam responds readily to treatment with lime, two sets of experiments were run with this soil. In the first no lime was used except that furnished by this slag fertilizer, but in the second set of tests the soil was limed at the rate of 2 tons to the acre. This was done in order to make sure that any beneficial effect observed from the slag treatment was not entirely due to the basic character of this material.

In Tables VI, VII, VIII, and IX the results of these experiments are given. The weight of the untreated plants, or checks, is taken as 100; and the weights of the plants grown under similar conditions, but in soils treated with various potash and phosphatic fertilizers, are compared with this figure.

TABLE VI.—*Relative green weights of wheat plants grown for a period of three weeks in Carrington silt loam untreated and treated with various quantities of potassic and phosphatic fertilizers.*

Treatment.	Application per acre.		Relative green weights.	Average.
	K_2O.	P_2O_5.		
	Pounds.	*Pounds.*		
Check			100	100
Slag (33SB)	50	60	101	104
Do	100	120	104	
Do	200	240	107	
Potassium sulphate	50		100	106
Do	100		113	
Do	200		106	
Acid phosphate		60	101	107
Do		120	107	
Do		240	114	
Potassium sulphate and acid phosphate	50	60	110	109
Do	100	120	110	
Do	200	240	108	

TABLE VII.—*Relative green weights of wheat plants grown for a period of three weeks in Hagerstown loam untreated and treated with various quantities of potassic and phosphatic fertilizers.*

Treatment.	Application per acre.		Relative green weights.	Average.
	K_2O.	P_2O_5.		
	Pounds.	*Pounds.*		
Check			100	100
Slag (33SB)	50	60	101	105
Do	100	120	107	
Do	200	240	106	
Potassium sulphate	50		122	127
Do	100		129	
Do	200		130	
Acid phosphate		60	101	101
Do		120	102	
Do		240	101	
Potassium sulphate and acid phosphate	50	60	111	121
Do	100	120	116	
Do	200	240	136	

TABLE VIII.—*Relative green weights of wheat plant grown for a period of three weeks in Volusia silt loam, untreated and treated with various quantities of potassic and phosphatic fertilizers.*

Treatment.	Application per acre.		Relative green weights.	Average.
	K_2O.	P_2O_5.		
	Pounds.	*Pounds.*		
Check			100	100
Slag (33SB)	50	60	113	112
Do	100	120	114	
Do	200	240	109	
Potassium sulphate	50		124	116
Do	100		117	
Do	200		107	
Acid phosphate		60	109	117
Do		120	113	
Do		240	128	
Potassium sulphate and acid phosphate	50	60	116	115
Do	100	120	119	
Do	200	240	109	

TABLE IX.—*Relative green weights of wheat plants grown for a period of three weeks in limed Volusia silt loam, untreated and treated with various quantities of potassic and phosphatic fertilizers.*

Treatment.	Application per acre.		Relative green weights.	Average.
	K_2O.	P_2O_5.		
	Pounds.	*Pounds.*		
Check			100	100
Slag (33SB)	50	60	104	116
Do	100	120	110	
Do	200	240	135	
Potassium sulphate	50		99	114
Do	100		142	
Do	200		100	
Acid phosphate		60	129	123
Do		120	124	
Do		240	116	
Potassium sulphate and acid phosphate	50	60	110	110
Do	100	120	118	
Do	200	240	103	

Tables VI, VII, VIII, and IX show that in every case applications of the slag fertilizer to the soils had a stimulating and beneficial effect.

The increase in growth caused by the citric soluble potash and phosphoric acid in the slag was, as a rule, less than that caused by the application of the equivalent quantities of these two fertilizer elements in a water-soluble form, but this is to be expected in the case of experiments carried on for such a short period of time. The tests, of course, are not conclusive, but they indicate that good results may be expected from the use of such a fertilizer.

SUMMARY.

A method of obtaining both potash and phosphoric acid in citric soluble form has been devised. It consists of mixing together phosphate rock and feldspar with the addition of small quantities of the oxides of iron and manganese to promote fluidity or lower the melting point of the slag, the mass being then heated to about 1,400° C. for about 20 minutes. The resulting product is not only soluble in a 2 per cent citric acid solution, but is also fairly soluble in water saturated with carbon dioxide. Pot tests with typical soils showed that the mineral increased the growth of wheat plants, but the beneficial effect derived from such applications was not, on the whole, as marked as it was when more soluble forms of phosphate and potash were used. The indications are, however, that the slag product has a distinct high fertilizer value.

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